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Tire-Wear Particles as Potential Ice-Nucleating Agents in the Atmosphere

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ABSTRACT: Tire-wear particles, generated through tire abrasion during driving, represent one of the largest global sources of microplastic pollution, with current annual emissions approaching 6 Tg. Due to their small particle sizes and low mass density, tire-wear particles can become airborne, undergo long-range transport, and potentially influence atmospheric processes. One critical but poorly understood pathway involves the heterogeneous freezing of supercooled cloud droplets, a key process in cloud glaciation and climate regulation. Here, we systematically investigate the ice-nucleating properties of laboratory-generated particles from summer, all-weather, and winter tires. Using optical microscopy as well as Fourier transform infrared (FT-IR) and Raman spectroscopy, we obtained particle-size distributions (∼100 μm mean diameter) and characterized the surface chemical compositions, confirming close similarity to the pristine tire materials. Ice-nucleation experiments performed with our custom-built IceBox instrument demonstrated that all tire particles consistently elevated the freezing temperatures of supercooled water droplets. The ice-nucleation performances of tire particles are found to be between feldspar and quartz, which are important mineral-based ice-nucleating agents in the atmosphere. Comparable results across all tire types suggest that major components such as rubber polymers or graphitic fillers are responsible for the observed activity. These findings establish tire-wear particles as effective atmospheric ice-nucleating agents, providing a baseline for future studies of environmentally aged tire particles and their potential roles in affecting the climate.

KEYWORDS: *microplastics, ice, climate, nucleating agents, heterogeneous nucleation, crystallization*

INTRODUCTION

Tire-wear particles are a major source of microplastics generated primarily through the abrasion of tires against road surfaces during driving.[1] About 3 billion new tires are produced annually[2,3] and the total annual global emission of tire-wear particles has been estimated to be close to 6 Tg.[4] The United Kingdom has been suggested to emit 79.5 Gg yr^{-1} of tire particles.[5] During the lifetime of a tire, around 10−16% of its weight is estimated to be converted into tire-wear particles.[6,7] Due to the global scale of road transportation, tire-wear particles are thought to be one of the largest sources of microplastic pollution in the environment.[1,8−11] The increased usage of electric vehicles, which are typically heavier than conventional combustion-engine cars and can exert more torque onto the tires, is expected to increase the global pollution from tire-wear particles in the future.[12−14] The pollution of the natural environment with tire-wear particles is considered to be a complex ecological and health hazard.[11,15−18]

Tire-wear particles can easily become airborne which is aided by their low mass densities.[19,20] Airborne tire-wear particles have been sampled directly from air, rainwater, and collected in remote locations following extended transport processes and residence times in the atmosphere.[19−23] The environmental impact of these airborne tire-wear particles in the atmosphere is unclear at present but potentially very concerning.

The freezing of supercooled water droplets in clouds is a fundamentally important process that dramatically impacts on the radiative properties of clouds, their lifetime and hence the general heat balance of our planet.[24−27] In essence, Earth's current climate relies strongly on the existence of supercooled water droplets in clouds. For clouds at lower altitudes, glaciation is typically caused heterogeneously by solid particles present in the atmosphere.[28] In the absence of ice-nucleating agents, water in the atmosphere can stay liquid at temperatures as low as −38 °C.[29] Naturally occurring ice-nucleating agents

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include mineral dusts[30−32] and biological materials.[33,34] Which sorts of particles can cause ice nucleation is still poorly understood at the fundamental science level and, therefore, difficult to predict.[35,36]

At present, the global distribution and number density of tire-wear particles in the atmosphere are not fully understood. Much of the current analysis therefore relies on the data obtained for microplastics in general of which the tire-wear particles are a major constituent.[11,23] The exact balance between contributions from terrestrial and marine sources toward atmospheric microplastics is debated.[37−39] Yet, there seems to be growing evidence that terrestrial sources are more important.[37,38] The current global atmospheric emission of microplastics has been estimated to range from 324 Gg to 8.6 Tg yr^{-1}.[23,37] Asia and the adjacent oceans alone may emit 310 and 60 Gg yr^{-1} into the atmosphere, respectively.[40] The terrestrial emissions appear to be linked to population density and poor waste management.[5,23,38] Microplastics have been modeled to be well-mixed throughout the troposphere and smaller particles are expected to reach the lower stratosphere.[41] Particles below 70 μm seem to be most relevant for atmospheric distributions processes.[23,38,42] Although microplastics are currently estimated to be a minor component of the total aerosol load of the atmosphere, as plastic production continues to accelerate and land-fill sources continue to degrade, atmospheric concentrations may reach levels where they significantly influence atmospheric processes in the future.[41] An incomplete understanding of atmospheric ice nucleation is a major source of uncertainty in climate predictions.[43] The question of the ice-nucleation ability of microplastics in general and tire-wear particles in particular therefore arises.

For this study, we prepared tire particles in the lab from commercially available summer, all-weather, and winter tires. The surface compositions of the tire particles were analyzed, and extensive ice-nucleation experiments were conducted with either tire particles immersed in water droplets or with water droplets in contact with films of tire particles.

MATERIALS AND METHODS

Preparation of Tire Particles

Three different commercial car tires were purchased from the same manufacturer including a summer tire (Michelin Primacy 4, 175/65 R15 H), an all-weather tire (Michelin CrossClimate, 145/60 R13 T), and a winter tire (Michelin Alpin 7, 175/60 R16 H). To produce tire particles, the tires were placed on aluminum foil and abraded using a nutmeg grater to collect several grams of material (Figure S1). The resulting particles were relatively coarse, so approximately one gram of material was transferred to a ceramic mortar and ground under liquid nitrogen, as shown in Figure S2. The particles broke with a characteristic crunching sound, and the grinding was continued until the pestle moved freely in a circular motion, and no further crunching was audible. The resulting fine tire particles were then carefully warmed to room temperature under moisture-free conditions and stored in glass vials. For comparison, thin slices were cut off of the tires using a sharp knife.

Characterization of Tire Particles

A Keyence VHX-970F optical microscope with a VH-Z500T objective was used to measure the diameters of the tire particles and to characterize particle shapes and morphologies.

Fourier transform infrared (FT-IR) spectra of the tire particles and thin slices were recorded using a Shimadzu IRTracer-100 FT-IR spectrometer. All tire materials were pressed firmly against the diamond window of the spectrometer's attenuated total reflectance (ATR) cell for the measurements. Raman spectra were obtained with a Bruker Senterra II spectrometer by using a 633 nm exciting wavelength and 10 mW laser power. Both the tire particles and the slices were positioned on glass slides, and the spectra were collected after focusing onto the samples using a 50-fold magnification objective.

Brunauer−Emmett−Teller (BET) physisorption data were obtained on a Quantachrome Autosorb IQ2 instrument using N_2 as a probe gas. Prior to measurement, samples were degassed at room temperature (~294 K) under high vacuum. Isotherms were measured volumetrically at 77 K using about 50 mg of sample.

Ice-Nucleation Experiments

Ice-nucleation experiments were performed using our purpose-built IceBox instrument described in detail in ref 44. The IceBox features a sample stage within a low-humidity environment that is cooled from 10 to −30 °C at a rate of 1 °C min^{-1}. A schematic and a photographic image of the IceBox are shown in Figure S3.

To prepare the sample stage, a thin layer of Vaseline petroleum jelly, which is known to exhibit poor ice-nucleation activity,[45−47] was applied. An array of 7 × 7 droplets of 1 μL Milli-Q water was then pipetted onto the stage, and the freezing processes were followed with an optical camera. Under these conditions, the average temperature at which half of the droplets froze, $T_{1/2}$, was −21.93 ± 0.44 °C for a cooling rate of 1 °C min^{-1}. This $T_{1/2}$ value serves as the baseline for this study, with any ice-nucleating effects of the tire particles expected to result in higher (less negative) $T_{1/2}$ values.

To disperse the tire particles in water, 0.5 g of the fine particles were stirred in 30 mL of Milli-Q water for 1 week using a magnetic stirrer. Droplets of these dispersions were then pipetted onto the Vaseline-coated sample stage, as previously described. Alternatively, Vaseline-coated sample stages were "dusted" with tire particles outside the IceBox by using a stainless-steel sieve. The fully coated surfaces were then mounted in the IceBox and water droplets were pipetted onto the thin film of tire particles. All ice-nucleation experiments were performed in triplicate or more to ensure reproducibility.

RESULTS AND DISCUSSION

Our experimental preparation procedure, which combines coarse abrasion with low-temperature grinding, produces gram-scale quantities of tire particles. These particles exhibit highly irregular shapes and rough, undulated surfaces, often with small protrusions and internal cavities, as shown in Figure 1a. These appearances are consistent with environmental tire-wear particles.[48−50] To determine the particle-size distributions (PSDs), the shortest diameters passing through the centers of the particles were measured, as indicated by the double-headed arrows in Figure 1a. The resulting PSDs, obtained from measurements of 250 particles from each of the summer, all-weather, and winter-tire samples, are presented in Figure 1b−d. For the summer-tire sample, 50% of the particles had diameters in the 0−90 μm range, whereas 50% of the all-weather and winter-tire particles fell within the 0−70 μm range. The average diameters were 100.9, 86.9, and 88.3 μm for the summer, all-weather, and winter tires, respectively, as indicated by the dashed vertical lines in Figure 1b−d. Environmental tire-wear particles have been reported to exhibit average diameters around 100 μm,[49,51,52] which aligns well with the sizes of our samples. As mentioned earlier, for atmospheric transport processes, particles below 70 μm seem to be most relevant.[23,38,42]

From a chemical perspective, tires are highly complex composite materials. Their primary components are natural (polyisoprene) and synthetic rubbers, such as styrene−butadiene and polybutadiene.[53,54] Common fillers include carbon black and silica, while additional additives typically comprise plasticizers, antioxidants, antiozonants, waxes, heavy

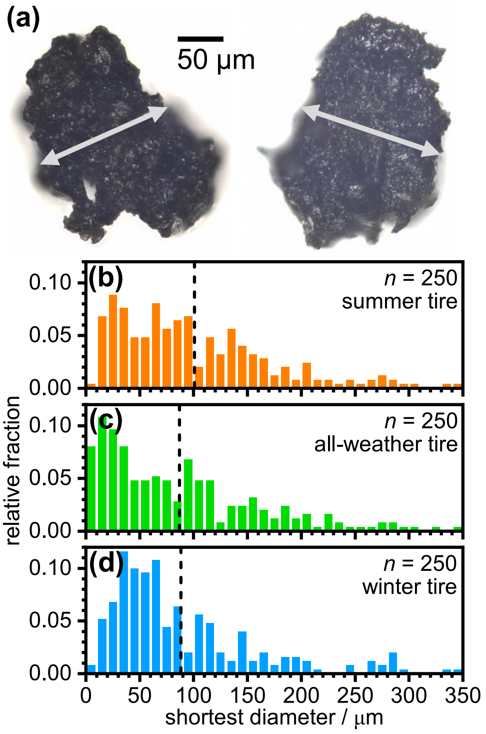

Figure 1. Particle shapes and diameters of lab-made tire particles. (a) High-resolution optical microscopy images of two larger tire particles. The two double-headed arrows indicate the shortest diameters measured through the centers of the particles. The particle-size distributions using the shortest diameters of 250 particles are given for tire particles produced from (b) summer, (c) all-weather, and (d) winter tires. The average diameters are indicated by dashed vertical lines.

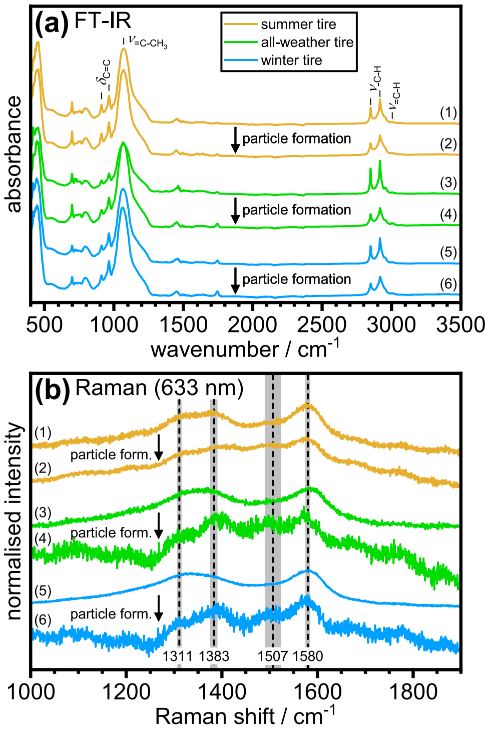

Figure 2. Chemical compositions of the as-received tires and the tire particles as seen with vibrational spectroscopy. (a) FT-IR and (b) Raman spectra of the as-received summer, all-weather, and winter tires are shown as spectra (1), (3), and (5). Spectra (2), (4), and (6) were recorded from the corresponding tire particles. The FT-IR spectra (a) are shown as recorded. The Raman spectra in panel (b) were normalized with respect to the intensity of the G-band. The average positions of the four peaks in the Raman spectra are indicated by dashed vertical lines, and the standard deviations are shown by the gray-shaded areas. All spectra in panels (a, b) were shifted vertically for clarity.

metals, and vulcanization agents.[2,53−55] The exact compositions will, of course, vary between different manufacturers. Overall, tire materials are inherently resistant to natural degradation, a property that is also expected for environmental tire-wear particles.

FT-IR and Raman spectroscopy analyses indicate that the chemical compositions of the surfaces of our tire materials change only minimally during particle formation using our preparation procedure. Figure 2a shows that the peak positions of all three tire types in FT-IR spectroscopy remain essentially unchanged after particle formation. The peaks just below 3000 cm^{-1}, associated with the stretching modes of aliphatic C−H bonds,[48,56] broaden slightly during particle formation, indicating a greater diversity of structural environments in the tire particles. The strong peaks at around 1070 cm^{-1}, attributed to the stretching or wagging of methyl groups adjacent to double-bonded carbon groups,[57] appear to increase in intensity during particle formation. This likely reflects a minor increase in the level of structural disorder around the methyl groups, which enhances the IR activity of these vibrational modes.

The Raman spectra shown in Figure 2b indicate the presence of graphitic species in the tire samples. The G-band at around 1580 cm^{-1} originates from the C═C stretching mode whereas the lower-frequency D-bands are typically associated with defects in graphitic materials.[58,59] To obtain accurate peak positions, the Raman spectra were fitted with four Pseudo-Voigt profile functions. The dashed vertical lines in Figure 2b show the average peak positions from all of the samples. The standard deviations of the peak positions, which reflect the differences in peak positions between the different samples, are indicated by gray-shaded areas. The D-bands are centered at 1311, 1383, and 1507 cm^{-1}, which means that they can be tentatively assigned to D4, D1, and D3 bands.[59] The peak position of the D4 band deviates quite a bit from the literature value of 1180 cm^{-1}.[59] As observed for the FT-IR spectra, no significant peak shifts are observed during particle formation and the relative intensities of the G to D-bands remain approximately constant indicating that no additional defects are introduced upon particle formation.[59] The noise levels in the spectra increase during particle formation, which is attributed to stronger light scattering from the small particles. Furthermore, the D-bands seem to become somewhat more structured after the particle formation, in particular for the all-weather and winter tires. This may result from a greater exposure of graphitic fillers on the surfaces of the tire particles, as they break down into smaller fragments.

Using our IceBox instrument, the three tire-particle types were found to raise the crystallization temperatures of supercooled water droplets. Within the IceBox, the freezing temperatures of supercooled water droplets are determined by cooling a sample stage at 1 °C min^{-1} as described in the Materials and Methods section and in detail in ref 44. The ice-nucleation properties of the tire particles were investigated by using two different experimental configurations: In immersion mode, the tire particles were first dispersed within liquid-water droplets and then positioned on nominally inert Vaseline films[45−47] on top of the sample stage. A schematic illustration of the immersion-mode configuration as well as a photo of the sample stage with 49 droplets of a tire-particle dispersion are shown in Figure 3a. Obtaining stable dispersions of the tire particles in water proved challenging as rapid agglomeration and precipitation were consistently observed. Neither ultrasonication nor high-shear mixing yielded satisfactory improvements. However, stirring the tire-particle dispersions with a magnetic stirrer for 1 week led to significantly more stable dispersions for all three types of tires. The effect of the prolonged stirring may be the reduction of electrostatic interactions between the tire particles and it may help to fully wet the rough, topographically complex surfaces of the tire particles thereby making them less hydrophobic. The immersion-mode freezing is of particular importance for mixed-phase clouds.[60]

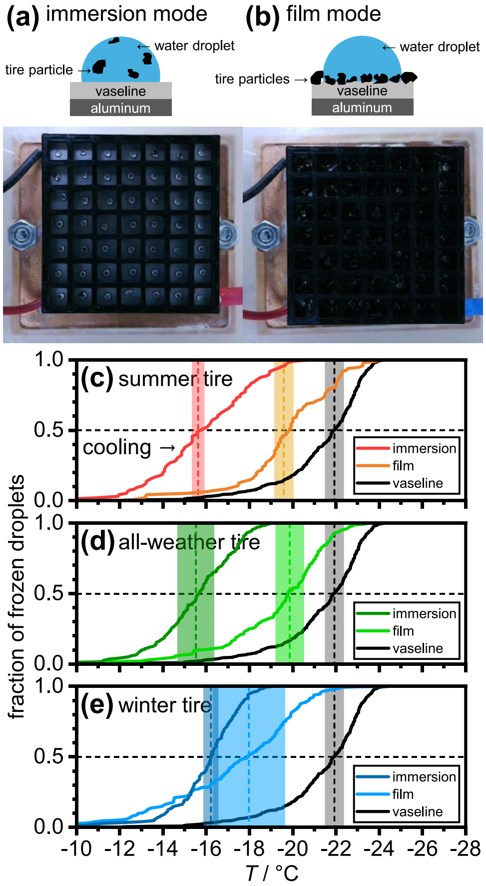

Figure 3. Testing the ice-nucleating properties of tire particles. Schematic illustrations and photographic images of the sample stage used in (a) immersion or (b) film mode with respect to the tire particles and water droplets. Fraction-frozen curves recorded upon cooling at 1 °C min^{-1} of the (c) summer, (d) all-weather, and (e) winter-tire particles either immersed in 1 μL water droplets or with the water droplets on top of films of the tire particles. The fraction-frozen curves using water droplets on Vaseline films are shown as black lines. The $T_{1/2}$ temperatures for the various samples are indicated by vertical dashed lines. The shaded regions are estimates of the experimental error of $T_{1/2}$.

For the film-mode measurements, the Vaseline-coated sample stage was uniformly covered with a fine layer of tire particles by using a dusting procedure, after which water droplets were pipetted onto the modified surface for ice-nucleation measurements. A schematic illustration as well as photo of a sample stage used in film mode are shown in Figure 3b. Although difficult to quantify precisely, back-of-the-envelope calculations suggest the film mode presents the water droplets with a smaller surface area of tire particles compared with immersion mode.

The fraction-frozen curves in Figure 3c−e show the fractions of frozen water droplets upon cooling at 1 °C min^{-1}. Clearly, the presence of tire particles elevates the freezing temperatures of supercooled water droplets, confirming their role as effective ice-nucleating agents. A consistent trend is observed for all three types of tires in which immersion mode yields freezing temperatures higher than those of film mode. Within the experimental uncertainty, the $T_{1/2}$ temperatures, at which half of the water droplets have frozen, are similar across all three tire types in immersion mode, with measured values of −15.63 ± 0.29 °C, −15.52 ± 0.86 °C, and −16.22 ± 0.35 °C for the summer, all-weather, and winter-tire particles, respectively. In contrast, the corresponding $T_{1/2}$ values for the film-mode experiments were −19.59 ± 0.45 °C, −19.86 ± 0.66 °C, and −17.97 ± 1.67 °C. These findings indicate that the way the tire particles interact with the water droplets exerts a stronger influence on the ice-nucleation temperatures than the specific type of tire.

Although the water droplets are nominally exposed to a smaller number of tire particles in film mode, part of the available surface area may also be obstructed by the underlying Vaseline film. Moreover, the difficulties encountered in dispersing the tire particles in water suggested that the wetting of the tire-particle surfaces is a slow process. The incomplete wetting may therefore be an additional factor explaining the colder freezing temperature observed in film mode.

Having established that tire particles can act as ice-nucleating agents, the question arises as to which tire component is responsible for this activity. The observation that summer, all-weather, and winter tires exhibit similar ice-nucleating properties suggests that minor, formulation-specific components are unlikely to be responsible. Instead, the ice nucleation is more plausibly attributed to one of the major constituents common to all tire types, such as the rubber matrix or the graphitic filler. Notably, various graphitic carbon nanomaterials have also been reported to act as ice-nucleating agents.[61−64]

To determine the ice-nucleation site density, n_s, of the tire particles, it is necessary to know their surface areas.[61,65,66] Using Brunauer−Emmett−Teller (BET) physisorption measurements with N_2 gas, only small uptakes of N_2 were found which makes the determination of accurate surface areas challenging. In general, using N_2 as the adsorbing gas, BET analysis is expected to work well for surface areas down to about 1 m^2 g^{-1} which can therefore be regarded as an upper bound of the surface areas of the tire particles.[67] A lower bound of the surface areas can be obtained by using the diameter distributions shown in Figure 1b−d and assuming spherical shapes of the tire particles. This approach gives 0.029, 0.028, and 0.024 m^2 g^{-1} for the summer, all-weather, and winter-tire particles. Considering the undulated, rough nature of the surfaces of the tire particles, as can be seen in Figure 1a, and the upper bound from the BET analysis, it seems reasonable to assume that the actual surface areas fall within the 0.1 to 1 m^2 g^{-1} range. Using this surface-area range and the fraction-frozen curves in Figure 3 from the immersion-mode experiments, n_s curves were calculated for the three types of tire particles using

$$n_s(T) = \frac{-\ln(1 - F(T))}{A} \tag{1}$$

where A is the surface area of the particles per droplet, calculated using the known droplet volume, and $F(T)$ is the fraction-frozen data as a function of temperature.[61,65,66,68] The results of these calculations are listed in Figure 4. As expected from the similar fraction-frozen curves and dispersion properties of the three types of tire particles, the calculated n_s curves are essentially on top of each other. For comparison, Figure 4 also shows the n_s curves of two important mineral-based atmospheric ice-nucleating particles, potassium feldspar and quartz.[31,69−71] On the basis of these data, it is possible to conclude that tire-wear particles show ice-nucleation performances between potassium feldspar and quartz.

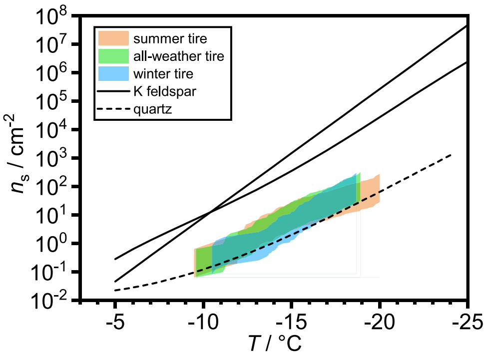

Figure 4. Ice-nucleation site densities, n_s, of tire particles as a function of temperature. The shaded regions show the possible n_s values calculated by using the immersion-mode fraction-frozen curves and surface areas ranging between 0.1 and 1 m^2 g^{-1}. The corresponding n_s curves for potassium feldspar and quartz were taken from refs 30,71.

In mixed-phase clouds, primary ice forms mainly by immersion freezing, with mineral dust as the dominant global source of ice-nucleating particles (INPs). Various atmospheric modeling studies have assessed the relative importance of the different ice-nucleating species in the atmosphere, taking into account aerosol distribution and loading. Potassium feldspar (K feldspar) is the most active dust component[30,71] and often controls glaciation in midlevel mixed-phase clouds.[72] Quartz, although ∼100× less active per unit area, is abundant so can still supply a substantial proportion of dust INPs and even dominate in some regions such as Patagonia, South Africa, and Western Australia.[73] Over oceans, marine organic INPs internally mixed with sea spray provide background INP levels at around $\sim 10^{-3}$ L^{-1} at −20 °C and often dominate in the Southern Ocean.[72,74] Black carbon INPs are generally thought to be minor contributors.[72] Warm temperature INPs ($\gtrsim -15$ °C) are often explained by dust carrying biologically derived macromolecules,[72] which are known to be capable of nucleating ice at warmer temperatures.[75]

Emissions of microplastics into the atmosphere (0.3 − 8.6 Tg yr^{-1})[23,37,40] are lower than those of minerals dusts (∼2000 Tg yr^{-1})[76] and sea-spray aerosol (∼5000 Tg yr^{-1}).[77] Yet, compared to mineral dust, microplastics have significantly lower mass densities,[19,20] and their emissions are more closely connected to areas with high population densities or poor waste-management systems.[5,23,38] Our work shows that tire particles have ice-nucleation abilities at least comparable to quartz. So it is possible that these differences are significant enough to make tire-wear particles the dominant INPs in some locations and under certain atmospheric conditions. It should also be mentioned that current detection techniques, and in particular, those used in the field, may miss microplastics smaller than a few micrometers in diameter which could mean that the actual microplastic emissions may be far greater than currently estimated.[78,79] Undoubtedly, there is a consensus that more field data, advanced characterization techniques and computer modeling are needed to fully understand the atmospheric plastic cycle including tire wear.[37,39,80]

In conclusion, our experimental procedure produces gram-scale quantities of tire particles, which would be challenging to collect directly from the environment. Moreover, environmental samples are typically heavily contaminated or even "encrusted" with road dust, paint debris, organic matter, and biofouling.[51] Furthermore, atmospheric transport processes can alter the chemical composition of tire particles through oxidation, ozonolysis, or photochemistry.[21] Consequently, environmental tire-wear particles exhibit significant chemical complexity, all of which may influence their ice-nucleation properties. For these reasons, we chose to perform "baseline" experiments using laboratory-prepared tire particles that retain the surface composition comparable to the corresponding pristine tires. We established that these can initiate ice nucleation, and this activity was attributed to one of the main components of the tires such as the rubber or graphitic filler. In this context, it should be mentioned that other types of microplastics have recently been shown to act as ice-nucleating agents as well including polyethylene, polypropylene, polystyrene, polyvinyl chloride, and polyethylene terephthalate microplastics.[81−84] In the case of polypropylene and polystyrene microplastics, it has been shown that exposure to sunlight conditions increases the ice-nucleation activities.[83] On the other hand, for polyethylene, polypropylene, and polyethylene terephthalate, aging of the microplastics was found to decrease their abilities to nucleate ice.[82,84] Very recently, micronization and nanoporosification of microplastics have been shown to increase their ice-nucleation activity.[85]

Having established that pristine tire particles can initiate ice nucleation, we now have a foundation for exploring the more complex behaviors of environmental samples. To fully assess the atmospheric impact of tire-wear particles, a comprehensive database will be needed, covering the ice-nucleating properties of all environmentally relevant microplastics and the effects of various aging processes. Combined with an accurate knowledge of the atmospheric number densities of tire-wear particles, this will enable estimates of whether, how, and perhaps most importantly, given the accelerating pollution, when they will influence cloud lifetimes and, ultimately, the climate.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsestair.5c00359.

> Photographic images of the tire-particle abrasion (Figure S1) and low-temperature grinding procedure (Figure S2); and additional details on the IceBox instrument (Figure S3) (PDF)

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Notes

The authors declare no competing financial interest.

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